MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

REPORT DOCUMENTATION PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
AFIT/CI/NR 85-141T		

4. TITLE (and Subtitle)	5. TYPE OF REPORT & PERIOD COVERED
In Vitro Comparison Of Zinc Phosphate And Glass Ionomers Ability To Inhibit Decalcification Under And Adjacent To Orthodontic Bands	THESIS/DISSERTATION
	6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)	8. CONTRACT OR GRANT NUMBER(s)
Donald J. Copenhaver	

9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
AFIT STUDENT AT: Temple University	

11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE
AFIT/NR WPAFB OH 45433 - 6583	Aug 1985
	13. NUMBER OF PAGES
	36 + figures

14. MONITORING AGENCY NAME & ADDRESS(If different from Controlling Office)	15. SECURITY CLASS. (of this report)
	UNCLASS
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

AD-A160 881

16. DISTRIBUTION STATEMENT (of this Report)

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

APPROVED FOR PUBLIC RELEASE: IAW AFR 190-1

LYNN E. WOLAVER 25 Sep 85
Dean for Research and
Professional Development
AFIT, Wright-Patterson AFB

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

ATTACHED

DD FORM 1473 EDITION OF 1 NOV 65 IS OBSOLETE   UNCLASS
1 JAN 73

ABSTRACT

IN VITRO COMPARISON OF ZINC PHOSPHATE AND GLASS IONOMER CEMENTS

ABILITY TO INHIBIT DECALCIFICATION UNDER ORTHODONTIC BANDS

Donald J. Copenhaver
Temple University, Philadelphia, Pennsylvania, 1985

Forty sets of impacted third molar teeth were banded with custom fitted
bands and cycled in a latic acid solution for four weeks. This solution
simulates an environment that would be found only in patients with the worst
oral hygiene. Each set was from the same patient. Visual, spectrophotometric
and pH testing were done on the sample sets. All areas not banded showed
significant decalcification after four weeks in the 0.10M latic acid solution.
The teeth banded with glass ionomer cement showed significantly less visible
changes under 10X dissecting microsopy than the zinc phosphate cemented
samples. Spectrophotometric analysis confirmed that significantly more calcium
was present in solution in the zinc samples than in the glass ionomer samples.
The significantly higher pH of the zinc phosphate cemented sample solutions also
confirmed more $Ca^{(++)}$ ions in solution. The evidence suggests that glass ionomer
cement protects the tooth surfaces from decalification under and adjacent to
orthodontic bands significantly better than zinc phosphate cements. Further
study on solubility and setting time of this new glass ionomer cement is
necessary before routine clinical use can be considered.

85-141T

AFIT RESEARCH ASSESSMENT

The purpose of this questionnaire is to ascertain the value and/or contribution of research accomplished by students or faculty of the Air Force Institute of Technology (AU). It would be greatly appreciated if you would complete the following questionnaire and return it to:

AFIT/NR
Wright-Patterson AFB OH 45433

RESEARCH TITLE: In Vitro Comparison Of Zinc Phosphate And Glass Ionomers Ability To

Inhibit Decalcification Under And Adjacent To Orthodontic Bands

AUTHOR: Donald J. Copenhaver

RESEARCH ASSESSMENT QUESTIONS:

1. Did this research contribute to a current Air Force project?

( ) a. YES   ( ) b. NO

2. Do you believe this research topic is significant enough that it would have been researched (or contracted) by your organization or another agency if AFIT had not?

( ) a. YES   ( ) b. NO

3. The benefits of AFIT research can often be expressed by the equivalent value that your agency achieved/received by virtue of AFIT performing the research. Can you estimate what this research would have cost if it had been accomplished under contract or if it had been done in-house in terms of manpower and/or dollars?

( ) a. MAN-YEARS _____   ( ) b. $_____

4. Often it is not possible to attach equivalent dollar values to research, although the results of the research may, in fact, be important. Whether or not you were able to establish an equivalent value for this research (3. above), what is your estimate of its significance?

( ) a. HIGHLY   ( ) b. SIGNIFICANT   ( ) c. SLIGHTLY   ( ) d. OF NO
       SIGNIFICANT   SIGNIFICANT   SIGNIFICANCE

5. AFIT welcomes any further comments you may have on the above questions, or any additional details concerning the current application, future potential, or other value of this research. Please use the bottom part of this questionnaire for your statement(s).

_____
NAME   GRADE   POSITION

_____
ORGANIZATION   LOCATION

STATEMENT(s):

IN VITRO COMPARISON OF ZINC PHOSPHATE AND GLASS IONOMERS

ABILITY TO INHIBIT DECALCIFICATION UNDER AND

ADJACENT TO ORTHODONTIC BANDS

Donald J. Copenhaver

D.D.S., University of Texas Health Science Center at Houston

A Thesis Submitted to the Faculty of the

Department of Orthodontics in

Partial Fulfillment of the

Requirements for a Master of Science in Dentistry

Temple University School of Dentistry

Philidelphia, Pennsylavania

August, 1985

This Thesis for

Master of Science in Dentistry

by

Donald J. Copenhaver

Has Been Approved by the Following Members

of the Thesis Defense Committee

of

Temple University School of Dentristry

Pouroth Pamirnia D. M.D., M.S.

Eugene Cohen DDS, M.S.

Milton Jay Lande, DDS M S.

KHOSROW Azizi DMD MS

Ronald Grasser DDS MS.

TABLE OF CONTENTS

ACKNOWLEDGEMENTS

i

INTRODUCTION

Zinc phosphate cement has long been the standard in orthodontics for cementation of appliances. It has many desireable properties including high strength, good manipulative time and relative insolubility in oral fluids. Perhaps its greatest shortcoming is its lack of protection to the enamel surfaces it contacts. This lack of protection coupled with patient neglect often leads to decalcification under and adjacent to orthodontic appliances.

Glass ionomer cements have recently held promise of significantly reducing the problem of decalcification in orthodontics. While possessing many properties similar to or superior to zinc phosphate cement, these new cements also appear to leach significant amounts of fluoride to the enamel surfaces.

The paper compares the decalcification protection of these two cements in vitro.

REVIEW OF THE LITERATURE

Enamel demineralization occurring under orthodontic bands is of concern to orthodontists. Factors contributing to this decalcification include seal breakdown, inadequate strength and bonding of the cements, and their solubility in oral fluids.[1] Of course, oral hygiene is also an important contributing factor.

Many authors[2-4] have discussed the higher incidence of caries in patients undergoing orthodontic treatment. Areas of enamel demineralization are often observed after removal of orthodontic bands. Tillery and associates[4] noted that enamel decalcification is the initial step in the development of carious lesions.

Fluoride has anticariogenic properties, and this has led to its incorporation into drinking water, dentrifices, and mouthwashes. Fluoride has also been used or occurrs naturally in dental cements used for cementing orthodontic bands.[5]

Recently, a new glass ionomer cement, incorporating fluoride was introduced. This cements unique properties include bonding to both stainless steel and enamel, release of stannous fluoride ions, and relative insolubility in oral fluids[6].

According to the American Dental Association,[7] certain factors must be considered when analyzing cements. These are strength (compressive and tensile), setting time, film thickness, solubility, adhesion, caries protection, pupal reaction, opacity, reflectivity, and color stability.[8] In orthodontics, the first six factors are all important, An ideal cement would have great

2

strength, ideal setting time (long manipulative time and rapid set), low film thickness (high wetability), be impervious to oral fluids, adhere to enamel and stainless steel, and impart caries protection to the contacted enamel surface.[7]

Zinc Phosphate Cement

Zinc phosphate cement powders consist primarily of calcined zinc oxide and magnesium oxide in the approximate ratio of 9 to 1.[9] The chief difference in cements appears to be in the composition of the powders. The variations occur in the addition of small amounts of modifiers, such as silica, rubidium trioxide, and bismuth trioxide.[10]

Zinc phosphate cement liquids are phosphoric acid solutions buffered by aluminum salts, and in some instances by both aluminum and zinc salts.[8] These metallic phosphate salts reduce the reaction rate of the liquid and the powder. The average water content of the liquid is 33± 5 per cent.[11] The amount of water present is a factor in the ionization of the liquid, and it is a critical ingredient in the rate of the liquid-powder reaction.[11]

The exact nature of the product of this reaction is not certain. Set cement contains no oxy-salts, so the term "zinc-oxyphosphate cement" is incorrect.[9] It has been generally thought that the tertiary zinc phosphate crystal $\{Zn_3(PO_4)_2 \cdot 4H_2O\}$, hopeite, is the final product. However, it has been reported by Servais and Cartz,[11] that no crystalline component forms during the setting process. The compounds formed by the reaction of the powder and liquid are non-crystalline, amorphous phosphates of zinc, aluminum, and magnesium. These

3

phosphates form only part of the set cement; the rest is unreacted cores of larger powder particles, surrounded by the phosphate matrix.[10] Servais, et al.[11] claimed that even though no crystalline phosphate is involved in the setting process of the cement, there can be a subsequent growth of hopeite in the presence of excess moisture (relative humidity 100%). The hopeite crystals are held weakly to the cement surface, so that their presence reduces any adhesive properties of the cement.

The proportion of powder particles and phosphate matrix varies with the amount of powder incorporated into a given amount of liquid. The set cement having the minimum amount of matrix has the best values for physical properties and is most suitable for use in the mouth.[10] Consistency of the mix is also determined by the amount of powder incorporated into a given amount of liquid. Since most manufactures comply with the requirements found in the American Dental Association Specification No. 8 for Dental Zinc Phosphate Cement, it behooves the practitioner to use the recommended powder/liquid ratio and mixing instructions provided.[12]

Norman, Swartz, and Phillips[13] determined the pH of setting cements with microelectrodes. They found zinc phosphate cement to have an initial pH of 4.0. This acidic pH rose to approximately 6.0 in 1-1.5 hours. It remained at this level until, at 4 hours it again rose to approach neutrality in 24 hours. In a subsequent study, Norman, et al[14], stated that variation in the powder/liquid ratio of zinc phosphate cement has the most influence on pH. Thin mixes have a lower initial pH than thick

mixes, and they remain 0.5 to 1.0 pH unit lower even after several days. Cook and Horne[15] used a spectrophotometric method of determining the pH of zinc phosphate cement and found it to have a pH of 5.0 after 30 days. They claimed that their study supported previous reports that the powder does not react completely with the liquid, and that residual acidity, in some form is present in set zinc phosphate cement.

Zinc phosphate cement has long been known to cause possible harmful effects on tooth structure.[16,17] Seniff,[18] using 8% SnF_2 as a topical solution to the enamel surface, produced a marked reduction in the etching effects of zinc phosphate cement. Muhler,[19] found that the application of 10% aqueous SnF_2, prior to the cementation of orthodontic bands, gave a significant reduction in dental caries and inhibited enamel demineralization.

The incorporation of SnF_2 directly into zinc phosphate cement could result in comparable protection, and the clinically practicality could be dramatic. Gursin,[20] dehydrated a small portion of the cement liquid and replaced it with 30% SnF_2, which resulted in a zinc phosphate cement. He found the $SnF_2/ZnPO_4$ cement to be superior in solubility reduction of enamel.

Scull,[21] incoroporated SnF_2 into zinc phosphate cement to determine its effect on decalcification under orthodontic bands. He compared the effects of this cement to plain $ZnPO_4$ cement and to a third cement, which had SnF_2 added to the powder portion at a 2.0% level. All cements showed protection against solubility of enamel, with the 2.0% $SnF_2/ZnPO_4$ cement exhibiting the most protection. It should be stated that the original cement had fluoride in it from the manufacturer.

5

Ram, Gadalia, and Reisstein[22] were concerned about changes in the physical properties of zinc phosphate cement with stannous fluoride added. In testing 2.0%, 5.0%, and 10.0% $SnF_2/ZnPO_4$ cement for consistency, setting time, compressive strength, film thickness, solubility, and degree of disintegration, they found the 5.0% and 10.0% cements showed detrimental effects in all categories. However, the cement with 2.0% SnF_2 exhibited no significant change in physical properties. The authors curiously failed to report the method used to incorporate the SnF_2 into the $ZnPO_4$ cement.

The L.D. Caulk Company manufactures a zinc phosphate cement (orthocem) containing 10.0% SnF_2 in the powder portion. The production of this cement is aimed at convenient commercial use in orthodontics to prevent the deleterious etching effect of regular $ZnPO_4$ cement. Wei, et al.[23] conducted a simulated in vivo experiment to determine the fluoride uptake by enamel with this cement using plain $ZnPO_4$ cement as a control. The enamel treated with 10.0% $SnF_2/ZnPO_4$ cement showed a significant amount of fluoride concentration. This was even greater than the fluoride uptake of another group that received a five minute application of topical 10.0% SnF_2 before cementation with regular $ZnPO_4$ cement. Myers, et al.[24] (1973) banded 68 teeth with this $SnF_2/ZnPO_4$ cement and regular cement by the same manufacturer. The teeth were extracted after seven days for orthodontic reasons, and no apparent topographical change in the enamel was observed with either cement.

Sadowsky et al.[25] studied enamel fluoride uptake from ortho-

dontic cements and its effect on demineralization. He used two Zn_2PO_4 cements one of which contained 5% SnF_2 and a third cement, a slicophosphate, all from the same manufacturer (Stratford-Cookson Company). He used enamel biopsies to test enamel uptake of fluoride under orthodontic bands. He then used the same biopsy technique to test the teeth after they had been subjected to a demineralization bath. He found that the cement containing SnF_2 gave its fluoride to the enamel surface, and this fluoride gave the surface significant protection against demineralization. Further, the greater the fluoride uptake, the more resistant the enamel was to demineralization. In a follow-up study,[26] he subjected teeth to a longer period in the demineralization bath and examined them micro-radiographically. He found that the enamel which had acquired fluoride fron the fluoride containing cements was apparently more resistant to caries attack.

Glass Ionomer Cement

Glass ionomer cements for restorative and luting purposes are based on the hardening reaction between ion-leachable glasses (fine Calcium aluminosilicate glass powders prepared with fluoride flux) and aqueous solutions of homopolymers and copolymers of acrylic acid.[7] The properties of the cement can be modified by using different formulations of glass and polyacid.[8] Working and setting time can also be varied by alteration of the size of the powder particle, addition of chelating agents such as tartaric acid, and adjustment of the concentration and viscosity of the liquid.[7] The setting reaction of glass ionomer cement is similar to silicate cements.[8] When the acid (acrylic acid co-

7

polymers) is mixed with the powders (fine aluminosilicate glass powders prepared with a fluoride flux), a paste is formed that rapidly hardens into a solid mass bound by a polysalt gel. These polysalts bind unreacted glass particles in a cement matrix. Wilson[28] regards formation of cement as an acid-base reaction between polymeric substances. The acid (the liquid) reacts with a base (the powder) to form a salt. Because of the polymeric nature of the salt, it acts like a binding matrix. The reaction is ionic, as are all salt forming reactions, that is, cations (+charge), interact with anions (-charge), to form a neutral salt.[28] Unlike the polymerization of restorative resins, no toxic monomers, initiators or activators are involved in the setting reaction. While setting, calcium and aluminum ions are displaced by the acid as positively charged ions, and fluorine is displaced as a negatively charged ion. Crisp and co-workers[29,30,31] have shown that the soluble calcium and aluminum ions react with negatively charged polycarboxylate ions in the liquid to form insoluble calcium and aluminum polycarboxylate. These are termed ion-exchange glasses, because the organic binding matrix is formed during the mixing of the cement.

The glass ionomers then, are similar to silicate cements. Calcium polycarboxylates are formed first as a firm gel that has the property of being carvable like amalgam. At this stage, the ionomer system is highly suspectable to water-sorption. If the water is absorbed, the matrix becomes chalky and can erode rapidly.[8]

The mechanism of adhesion of glass ionomer cement has yet to be fully explained. Glass ionomer cements can bond only to sur-

faces with which they can chemically react and that provide cations (dentin is the one exception)[32]. Adhesion results from electrostatic attraction with cations sandwiched between negatively charged oxide surfaces and COO$^-$ groups in the cement.[32] These mechanisms can only operate effectively if intimate contact is achieved between the adhesive and the tooth substrate: such contact is the first requirement for good adhesion.[28] Thus the adhesive is required to spread over the tooth surface displacing contaminants (i.e. water and air). Unlike other cements, glass ionomer cements also adhere to dentin. Collagen contains some pendant chains that include COOH and NH$_2$ groups. These could provide sites for adhesive ionic and dipole interactions.[34] Because the attachment is by ionic and polar bonds, the release of fluoride and its uptake by the enamel are facilitated.[34] The intimate molecular contact facilitates exchange of the fluoride ions with the hydroxyl ions in the apatite of the surrounding enamel.[34] The intimate contact from the molecular interaction is essential for flouride ion-exchange. In contrast, a material that does not adhere by molecular interactions would leave gaps between the cement and tooth, so that, even if it releases fluoride, ion-exchange would be hindered (i.e. zinc phosphate cements with fluoride).

Glass ionomer cements adhere to enamel and dentin in a manner similar to carboxylate cements.[8] Several studies have shown that when precious metals are electroplated with one to two microns of tin, the ionomer cement will adhere to the surface of tin oxide via polar and ionic bonds[32,34].

9

McClean and co-workers[36] have shown that the matrix of the glass ionomer cement contains sheathed droplets of calcium fluoride that confer the ability to leach fluoride ions into the surrounding tooth enamel. Kidd[37] using an artifical caries technique with a diffusion controlled acidified gel, concluded that ionomer cements exert a cariostatic effect. Tveit and Gjerdet[38] found that fluoride release from a glass ionomer cement was five time less than from a silicate cement. However, they were the only workers to report this difference. In fact, Cranfield,[39] in testing four glass ionomer cements, found that the release rates of glass ionomers and slicates are broadly comparable. He also found: that after thirteen weeks there was no sign of deviation from linear fluoride release of glass ionomer cement, and that the fluoride release seemed to derive from the entire volume of the cement. Others found similar results in their studies.[40,41] Sylvia et al.[40] tested three glass ionomer cements for fluoride release and stated that powder/liquid ratios influence the release rate and total release of fluoride from these cements: a higher powder:liquid ratio reduces solubility due to the lower proportion of liquid which promoted rapid dissolution. Retief et al.[42] testing fluoride uptake from a glass ionomer cement by enamel and cementum, found that the fluoride acquired by the enamel and cementum was largely due to the topical effect of the released fluoride in {synthetic} saliva. They concluded that enamel and cementum can acquire <u>substantial</u> amounts of fluoride from a glass ionomer cement.

If the release of fluoride from glass ionomer cement is at least as much as silicate cement, then glass ionomer cement

should impart significant protection protection to enamel under or adjacent to orthodontic bands. De Fritas[43] found that fluoride-ion release from silicate cements was still occurring after fifty-two weeks at a rate substantial enough to protect adjacent enamel ($0.16ugm^{-1}g^{-1}$).

The major criticism of glass ionomer cements is their tendency to calkiness with early contamination from moisture.[8] A limited number of clinical studies have been done. In one in-vivo study, done with cement on patches over crowns, Michem[44] showed glass ionomer to be the least soluble when compared to polycarboxylate, silico-phosphate, zinc phosphate, zinc-oxide eugenol and EBA+alumina cements. Causton[45] studied earlier glass ionomers by exposing them to water during the first forty-eight hours after set. The degree of hydration, rate of fluoride release and degree of cross-linking of the gel-matrix were shown to be affected by the water ingress during the setting reaction. Oils[46] also found that the glass ionomer cements seemed to be sensitive to moisture during the setting reaction. Wilson and Crisp[47] found that adding chelating comonomers like tartaric acid could significantly increase the rate of hardening without reducing the working time (tartaric acid is the accelerator in ESPE Ketac-Chem). Pluim,[48] in another experiment, determined the in-vivo dissolution rate of glass ionomer to be only one-eightheeth (1/80) that of zinc phosphate cement. ESPE's glass ionomer system, has tartaric acid in the liquid as an acelerator. It reaches a hard set seven minutes after start of mixing. This is a significant improvement because, before this development, glass

ionomer systems had to be protected from water sorption for thirty minutes or rapid dissolution would occur.[49]

Compressive strength of glass ionomer cements is 4.5 N/mm^2, tensile strength is 247N/mm^2.[8] This compares to zinc phosphates 7N/mm^2 compressive strength and 83N/mm^2 tensile strength.[8]

METHODS AND MATERIALS

Rationale and Design:

This study was an attempt to elict the clinically significant caries protection difference between a glass ionomer and a zinc phosphate cement. To accomplish this, all experimental factors except the two cements were kept as constant as possible.

The teeth sample sets were from the same patient, all teeth were impacted, all teeth were stored in the same solutions, banded by the same operator on the same day, cycled in the same solutions, and examined both prior and after the experiment in a blind comparison by the same operator.

The following chart details the experimental design.

```
        60 Sets examined for evidence of decalcification
        on facial, lingual, mesial and distal surfaces
                            ||
                            \/
        40 sets selected on basis of no observable
        decalcification, same level of development, and
        acceptable morphology for banding.
               ||                                      ||
               \/                                      \/
    30 SETS: Regular mix of both          10 SETS: Regular mix of
    cements as per manufacturers            both cements as per
    instructions                           manufacturers instructions
               ||                                      ||
               \/                                      \/
    ZnPO4 sample cycled                   10 SETS: In individual
    in 610 ml of latic acid                   6cc vials to test for
    pH 4.5, .10 M                         Ca++ levels
                                                       ||
               +                                       \/
    Glass ionomer sample cycled           Spectrophotometric analysis
    in 610 ml latic acid solution         and pH determination of test
    pH 4.5, .10 M                         solutions         ||
                                                            \/
                                          pH determination of Zn2PO4
               ||                         and glass ionomer solutions
               \/                                      ||
                                                       \/
        40 sets examined for enamel surface changes.
```

Sample

Forty sets of freshly extracted human teeth were used. The teeth sets were gathered from oral-surgeons in the Philadelphia area and from the United States Air-Force Academy, Colorado Springs, Colorado. The teeth were all previously fully impacted with no portion of the crowns exposed to oral fluids. Sixty sets were examined and the best forty sets were chosen for the sample. All teeth samples had acceptable crown morphology for banding. To facilitate banding, the sample teeth roots were imbedded in orthodontic cold cure acrylic resin. Teeth from a single patient were placed on each block. The sets were numbered and placed in a solution of 35% ethyl alcohol and stored at room temperature prior to banding. Previous studies testing cements have used this storage solution.[25] The blocks containing the teeth sets were sectioned, the teeth carefully cleaned with flour of pumice in a slowly rotating rubber cup, then given a ten minute fluoride treatment with 10% acidulated fluoride gel. The exposed root surfaces were then coated with clear nail polish to avoid root suface demineralization. Figure 1 shows a typical tooth set ready for banding. Each tooth was examined under 10X dissecting microsopy (Bausch and Lomb, Stero-zoom dissecting microsope, with two B & L 31-35-38 illuminators), for evidence of enamel defects. These pre-decalcification readings were sealed in an envelope and not opened until the post experimental observations had been completed.

Cements

The cements tested were Ames Gray-brown zinc-phosphate cement, and ESPE Ketac-Chem glass ionomer cement. The cements were mixed according to the manufacturers instructions:

The zinc-phosphate cement was placed on a glass slab and mixed at room temperature by the timed addition of powder to liquid. The measuring system was developed by the author and consisted of a calibrated mixing scoop and a one ml syringe. The scoop was calibrated by small additions of cold cure acylic and subsequent weighing of the powder on both a O'Haus Cent-O-Gram accurate to 1/100 gm and a Mettler precision balance accurate to 1/1000 gm. The dispensing spoon was found to be accurate to±.05 gm by repeated weighings. The powder was fluffed and measured with the scoop. The recommended powder/liquid ratio was 1.3 gm powder to 0.5cc liquid. For this study the author used one-half this amount or .65 gm of powder and 0.25 gm of the liquid for cementing each band.

The glass ionomer was mixed by incorporating the powder into the liquid. The ESPE system comes with a measuring scoop and drop dispensor. These were tested by repeated weighings and found to be accurate enough to be used in this study (±.05gm). ESPE recommends seven minutes from the start of the mix to completion of set and exposure to oral fluids; Three minutes, thiry seconds working time, and three minutes, thirty

setting time. The recommended powder/liquid ratio is 3.4 to 1.

The mixing of the glass ionomer was done by fluffing the powder, measuring one level scoop of the powder, and mixing with two drops of the liquid from the dispensor. This produced the recommended powder/liquid ratio. Each band received a fresh mix.

Banding

Banding was done by a single operator using the following method:

1. All teeth sets were cleaned with water and flour of pumice in a slowly rotating rubber cup.

2. Each band was custom fitted using Unitek .004mm thick contoured molar band blanks.

3. Each band was then coated with cement and cemented to place using the band plier to snug the band.

4. Each band was then welded on the facial surface of the tooth before the cement set occurred.

5. Hand instruments were used to adapt the occulsal and gingival margins of the band to the tooth.

6. All sample sets were placed in the test media within eight hours of banding.

Figure 3. shows a sample tooth banded with a control tooth after decalcification.

Controls

Controls were two fold. First it was assumed that calcification levels for contralateral impacted teeth were generally

16

equal at the beginning of the experiment. This was assumed because the sets were not exposed to oral fluids. If differences in root length or other morphology were noted at the time of placing the teeth in blocks, then these sets were discarded.

In some instances, there were three teeth from a single patient. These extra teeth were mounted on the same blocks as the other two from that patient. These extra teeth were used as additional controls.

After the teeth were mounted on acrylic blocks, they were examined under 10X microscopy and graded according to the following "Enamel Changes Scoring System" (after Shannon, etal).[5]

   -Enamel Changes Scoring System-

   1. NONE: No color change evident

   2. MILD: Slight change in enamel color

   3. MODERATE: Definate whitening of enamel without break in continuity of surface.

   4. SEVERE: Obvious area of discoloration with roughness and break in continuity of surface.

   5. CARIES: Obvious break in continuity of enamel surface.-

All teeth were examined and graded according to the above scale. The location of existing color changes was noted as to surface: mesial, distal buccal, lingual. Two observations were made for each tooth surface. Figure 2. shows examples of the five levels of decalcification used for grading the visual observations in this study.

Cementing proceedure

Each of the two cements was measured as previously des-
cribed. The cements were mixed on a glass slab at room tempera-
ture. Each mix was timed to insure strict compliance of manufac-
turers instructions. All cements were mixed by the same opera-
tor. Each tooth recieved a fresh mix. After the recommended
setting time, the excess cement was removed with a scaler and the
samples were placed in the latic acid solution.

Testing Solution

The test solution selected was 0.10M latic acid solution
buffered to pH 4.5 with sodium hydroxide. This solution was used
by Grieve[51] to produce caries-like white spot leisons in vitro.
The teeth were in this solution for 27 days. The solution was
changed three times (every two weeks) except for the individual
teeth suspended in six ml. vials. These solutions were only
changed once because the spectrophotometric testing required a
certain concentration of calcium ions for accurate analysis. The
lower storage temperature used (7° C vrs 20° C) accounted for
the longer solution cycle time necessary to develop decalcifica-
tion on the enamel surfaces. The individual vials were cycled
an additional two weeks (41 days total) in their individual six
ml. vials. Two seperate samples of the soultions were taken from
these vials. The first set was taken after the initial test
period of 27 days. These samples were used in the spectrophoto-
metric analysis and for the initial pH measurements. Fresh solu-
tion was then added to the six ml. vials and they were stored in
this solution for an additional two weeks for a total of 41 days.

18

This was necessary because visual observation of the samples revealed a reduced decalcification of the controls suspended in this small volume of solution. The decision to terminate the cycling was done on the basis of observed demineralization of the exposed surfaces of the tooth samples and by observing the decalcification of the control teeth.

Method

All teeth were examined before the experiment under light microsopy, then banded as previously described. After a period of 27 days in solution, the sample teeth were washed, the bands carefully split with band removing pliers, and the remaining cement removed with a sharp instrument. All teeth were then re-examined twice by the same operator using 10X microscopy. The same grading system previously mentioned was used again. The solutions from the individually suspended sets were tested for the presence of calcium ions and the first pH readings were recorded. Two weeks later (41 days), a second pH reading was taken on the ten teeth subset.

Spectrophotometric Analysis

The ten samples suspended in individual six ml. vials were the sets used for analysis of Ca++ ions lost to their test solutions during the decalcification period. The spectrophotometric analysis was done using the Fisher Scientific Analysis Method (see appendix for complete test proceedure), with a Bauch and Lomb Spectronic 80 spectrophotometer operating at 540 Nm calibrated to a 10mg% calcium standard. This testing was done by an independent operator. This was a

19

blind comparison; the operator had no knowledge of the contents of each of the tested solutions.

PH Testing

PH determinations of all solutions were done at two times during the experimental period. The first reading was taken at 27 days, the second at 41 days.

PH determinations were done with solutions at room temperature with a Fisher Model 810 Accumet Digital pH meter calibrated to pH 4.0 at 15° C.

Each of the six ml. solutions were tested individually, with two samples (27 days and 41 days) from each vial. The 0.10M latic acid solution was checked each time to test for possible degradation of the test solution during the experimental period.

To check for possible inf ences of the cements themselves on the pH of the test solutions, equal samples of each cement were mixed, allowed to set completely and then suspended in 10 ml of the 0.10M latic acid solution for two weeks. These solutions were also checked for pH change against the unused test solution and the pH 4.0 standard.

The results are presented in three parts; visual observations with 10X light microscopy, pH differences in test solutions, and spectrophotmetric analysis for calcium of six ml. test solutions.

Visual Observations

The visual observations are presented in Tables 1., 2., 3. and 4. All samples showed significant changes during the experimental period. All banded teeth showed significantly less decalcification in the banded area compared to the unbanded controls. The differences between the teeth sets were observed generally at the occulsal and givgival band margins. Four surfaces (mesial, distal, buccal and lingual) were evaluated and the findings for these four surfaces were summed.

The purpose of the second observation was to check the repeatibility of the scoring method used in the visual observations. Observer errors between the first and second decalcification reading are presented in Table 8. There was an overall error of 6.5% for 640 observations. All errors were one scale increments. The most common observation error was between 1 (no color difference), and 2 (slight color difference). Because of the small observer error, the first observations were used for the statistical analysis.

Table I compares the differences between the teeth cemented

with glass ionomer before and after decalcification with the .10M latic acid solution. At the .01 confidence level, the differences were not significant. The average difference was 0.06 with a standard deviation of .20.

Table II shows the differences between the teeth cemented with zinc phosphate cement before and after decalcification. At the .01 confidence level, there were significant differences (T = 13.07) between the tooth surfaces. The average difference was 1.39, with a standard deviation of .66.

Table III compares the zinc phosphate to the glass ionomer samples before the experiment to assertain if the samples were the same at the beginning of the experiment. At the .01 level the samples showed no significant differences (t = .29).

Table IV shows the differences between the teeth sets after the experimental period. Significant differences appeared between the average tooth surfaces of the glass ionomer cemented teeth versus the average tooth surfaces of the zinc phosphate cemented teeth (t = 11.00 at the .01 level).

In order to access whether the ten teeth cycled in **individual** vials of the latic solution behaved differently than the teeth cycled together, averages and standard deviations were calculated for the set of thirty (without the individually cycled teeth) and the set of forty (including the individually cycled teeth). This data is presented in table V. As this table shows, no significant differences occurred between these two subsets, so the grouped data for the visual observations was used.

Tables I - V are summarized below and in table VI.

```
                     BEFORE CYCLING          AFTER CYCLING

GLASS IONOMER        avr = 1.038    ->       avr = 1.10
                     sd  = .20      ns       sd  = .20
                        |                       |
                       \|/          t = 1.95   \|/
                        ns                      sig
                     t = .29                  t = 11

                       /|\                     /|\
                        |                       |

ZINC PHOSPHATE       avr = 1.05     ->       avr = 2.44
                     sd  = .66      sig      sd  = .67

                               t = 13.07
```

avr = average
ns = not significant at the .01 level
sig = significant at the .01 level
t   = two tailed T test
sd = standard deviation

pH Differences

Two pH readings were taken for each cemented tooth in the ten sample subset. The first reading was taken at the end of the initial experimental period (27 days), and the second reading was done three weeks later when visual observation confirmed that the samples had decalcified roughly the same as the thirty sets cycled together (41 days in solution). The readings are presented in table VII. The differences between the pH readings of the glass ionomer and zinc phosphate cemented teeth solutions in the first reading averaged .68 pH units. This was a significant difference at the .01 confidence level (t = 7.3). The second pH reading produced similar results, the average difference being

To determine what affect the different cements themselves might have on the pH readings, equal set samples of the two cements were cycled in six ml. vials of the latic acid solution for four weeks. The pH readings before and after suspension in the solutions were roughly equal, the zinc phosphate cement pH reading was 4.78, and the glass ionomer cement pH reading was 4.64 pH units, the difference being .05 pH unit. The pH of the unused latic acid solution was 4.50 both at the beginning and end of the experiment. Figure 5 graphs the pH findings.

Spectrophotometric determination of calcium ion concentration

The findings for the calcium ion concentration of the individual teeth solutions are presented in table VII. Their were significant (t = 7.88) differences at the .01 confidence level between the teeth cemented with glass ionomer and zinc phosphate cements. The solutions containing the zinc cemented teeth had, on average, .210 mg% more calcium (S.D. = .08) in solution than the glass ionomer cemented teeth. Figure 6 graphs the calcium ion concentration differences between the teeth sample sets.

DISCUSSION

The findings show that there is a significant difference between zinc phosphate and glass ionomer cements ability to protect tooth surfaces from decalcification under and adjacent to orthodontic bands.

The severe environment, .01M latic acid solution, can arguably be said to be more severe than that found in the normal oral environment. However, Grieve[51] found this solution produced white spot leisions that most closely resembled those leisions found in the mouth. Whether or not this solution represents a reasonable facimile of conditions occurring in vivo is unknown. Previous studies on the oral environment by Fehr, Adams and Bloom [53-55] suggest that lactobacilli producing latic acid play a major role in enamel decalcification during orthodontic treatment.

The scoring method used for visual observation was partly responsible for the high correlation coefficient values obtained in the visual observation portion of this study. The five values (1 = no color difference, 2 = slight color difference, 3 = whitening, 4 = discoloration with whiteness, 5 = carries) are not a true linear progression. For example, the difference between a one score and a two score (100%) does not really correlate to a 100% change in the tooth surface. However, it was clear after examining the tooth surfaces that the glass ionomer cemented tooth surfaces were closer to their original morphology than the teeth cemented with zinc phosphate cement. Figures 7. and 8.

25

show a typical tooth set after decalcification. Figure 7. shows the dividing lines between the banded and non-banded areas of the zinc phosphate cement sample are indistinct with areas of decalcification extending in to the banded area. In contrast, the glass ionomer cemented sample (figure 8) shows a clear, sharp border between the banded and non banded areas of the tooth surfaces. The overall glassy surface appearance in the banded areas of the glass ionomer cemented sample is closer to the original surface appearance than the surfaces found on the zinc phosphate cemented surfaces. Many of the zinc cemented surfaces were characterized by white bands on the occulsal and gingival band margin areas consisteint with decalcification lines found in vivo. These lines generally corresponded with a score of 2 (slight color differnce) or 3 (whitening). The average score for the zinc phosphate cemented teeth of 2.44 was more than double the average score for the glass ionomer cemented teeth (1.10) in the post experiment observations (table IV). None of the glass cemented teeth but seven of the zinc phosphate cemented teeth had decalcification score average higher than 3. It should be remembered that all unprotected tooth surfaces were decalcified to the three or four level on the scale as were all of the control teeth surfaces. From this, it is seen that just the presence of the orthodontic band and either cement afforded substantial decalcification protection to the banded area of the tooth surfaces.

The areas where most differences between the tooth surfaces were observed were along the margins of the bands. It appeared that the seal integrity of the zinc phosphate cemented bands was

inferior to the seal integrity of the glass ionomer cemented bands. Areas of decalcification extending well beyond the band margins were also fairly common. This indicates that either the cements ability to seal the margins of bands differs, or the zinc cement is more soluble in the test solutions. Glass ionomer cement has been shown in vitro to adhere to stainless steel and to tooth enamel with a chemical bond[51]. Zinc phosphate, on the other hand, does not chemically adhere to enamel or stainless steel, though through partial etching of the enamel surface it forms an intimate association with the tooth surface.[12] The pH testing of the two cements did not show an appreciable difference between their solubility rates, though other studies have shown zinc phosphate to be more soluble in oral fluids than glass ionomer cement.[44]

Another factor that has to be considered is the tightness or "fit" of the experimental bands. Although every effort was made to achieve as tight a fit as possible, it is the authors opinion that these bands were not as tightly adapted as can be achieved in vivo. The reasons for this are two fold: First in using custom pinched bands, a uniform procedure had to be established to assure the same fit of every band. This necessitated a deviation from normal banding procedures. Second, the tooth samples were all third molars, and even though they were carefully selected for acceptable anatomy, many possessed more occulsal gingival taper than would normally be found. This made consistent banding more difficult and may have partially accounted for the increase in decalcification noted in the visual observations.

It was because of the necessarily subjective visual observa-

tion part of this experiment that the calcium ion level determination was done. This part of the study correlated with the visual observations. As was noted, the solutions of the ten sets of teeth cycled in six ml. of the latic acid solutions did show significant differences between the two cements. All of the teeth cemented with zinc phosphate cement showed more calcium in solution than the teeth cemented with glass ionomer cement. It should be remembered that these differences were from matched teeth sets from the same patient. They had not been exposed to any oral fluids. The average of .32mg% calcium in solution in the zinc cemented teeth was nearly triple the amount of calcium in solution in the glass ionomer cemented samples (.11mg% calcium). This indicates that for a given set of teeth, the glass ionomer cement was better at preventing loss of calcium to the decalcifying solution than the zinc cement. This could be due to superior sealing ability of the glass cement, but it probably is due to the ability of the glass cement to leach substantial amounts of fluoride to the surrounding teeth. This ability, combined with the chemical bonding to the tooth enamel could have been responsible for the differences observed. Figure 4 shows a glass ionomer set after debanding with its control tooth. The tooth in the foreground is the control tooth. The glass ionomer cement can be seen adhering to the sample after debanding. During debanding, the seal or bond was usually weakest between the band and the cement. Removal of this cement was more difficult than with the zinc phosphate cement. This could be a potential problem clinically, especially on the mesial and

28

distal surfaces.   The bond strength of glass ionomer is not as strong as Bis-GMA resins.   Caryle _et_ _al_ found that the bond strength of the glass ionomer cements was roughly one-forth that of composite resin systems.

The pH measurements correlate with the calcium ion measurements. The readings were done at the end of the 27 day initial experimental period and again two weeks later. The presence of calcium in soultion would make the solution more alkaline and raise the pH. The initial solution pH of 4.5 was checked at each pH reading. The solutions in which the zinc phosphate cemented teeth were suspended all had higher pH readings than the glass ionomer cemented teeth, indicating more calcium in solution. The test for possible interference from the composition of the cements themselves in the pH readings showed no significant pH difference between the two cements samples.

SUMMARY AND CONCLUSIONS

Forty sets of extracted human teeth were banded with two different cements and cycled in latic acid solution to test for differences in each cements ability to inhibit decalcification under orthodontic bands. Each set of teeth was from the same patient. After a period of 27 days, the sets were compared with pre-experimental visual observations for cahnges in the facial, lingual, mesial and distal surfaces. A subset of ten of the banded teeth which were cycled in their own six ml. vials were analyzed for the presence of calcium by spectrophotometric analysis. PH readings were also done on the ten teeth subset.

The results show that all of the banded surfaces were resistant to decalcification when compared to the nonbanded teeth. The glass ionomer banded teeth showed significantly less decalcification than the zinc phosphate banded teeth. The glass ionomer cemented teeth also had significantly less calcium in solution than the zinc phosphate cemented teeth. Further, the zinc phosphate cemented teeth solutions were significantly more basic, again indicating more calcium in solution than the glass ionomer cemented teeth.

The results indicate that glass ionomer has potential to be useful in cementing orthodontic bands, especially when decalcification is a major concern. It should be remembered however, that problems with glass ionomer cement have still to be resolved. The major problem is the seven minute setting time from

30

start of the mix during which the cement must be kept isolated from moisture contamination. This new cement is not forgiving in this regard. Any moisture contamination will seriously compromise the solubility and strength of the set cement.[34] Also, the American Dental Association, while approving use of the cement, has not specifically listed cementation of orthodontic bands as an indication for use of this new cement. Finally, little research has been done to test the validity of ESPES claim that Ketac-Chem's new formulation actually is completely stable after only seven minutes. All earlier formulations of this cement had to be protected from moisture contamination with a varnish for a minimum of 30 minutes after initial set. This of course would make glass ionomer not practical for orthodontics. Further research needs to be done to establish conclusively the solubility of this new cement formulation at various setting times before contamination with moisture. If this experiment proves the validity of ESPES claims, then an in-vivo study could be performed perhaps using contralateral teeth on the same patient and accessing decalcification clinically after orthodontic treatment.

Decalcification remains a problem in Orthodontics. If the results of this suggested research prove the stability of ESPES Ketac-Chem cement, then this new glass ionomer formulation offers the potential to significantly reduce decalcification in orthodontic practice. Possible uses for this cement could include recementation of loosened bands, cementation of bands where tooth morphology precludes a tight seal, cementation of long term

retention appliances such as banded lower retainers, and for cementation of maxillary molar bands in early treatment where long term wear of appliances is anticipated. The author recommends however, that routine clinical use of this cement in orthodontics be delayed until further studies are done.

APPENDIX

BIBLIOGRAPHY

1. Sadowsky, P.L., and Retief, D.H. 1976, A comparative study of some dental cements used in orthodontics. Angle Ortho 46: 171 - 181

2. Noyes, H.J. 1937, Dental caries and the orthodontic patient, J. Am. Dent. Assoc. 24: 1243 - 1254

3. Hughes, D.O., Hembrose, J.H., and Weber, F.N. 1979, Preparations to prevent enamel decalcification during orthodontic treatment. Am J. Ortho 75: 416-420

4. Tillery, T.J., Hembree,J.H., and Weber, F.N. 1976, Preventing enamel decalcification during orthodontic treatment. Am. J. Ortho 70: 435-438

5. Shannon, T.L., St Clair, J.R., Prult, G.A. and West D.C. 1977, Stannous fluoride verses sodium fluoride in preventitive treatment of orthodontic patients. Aust J. of Ortho. 5: 18-24

6. Maldonado, A., Swartz, M.L., Phillips, R.W. 1978, An in vitro study of certain properties of a glass ionomer cement. JADA 96: 785-796

7. Crisp, S. et al. 1975, Properties of improved glass-ionomer cement formulations. J. Dent. 3(3): 125-130

8. Smith, D.C. 1971, Dental Cements. Dental Clinics of North America. 15(1): 3-31

9. A.D.A. Guide to Dental Materials and Devices, Tenth edition, 1980-81

10. Phillips, R.W. Skinner's Science of Dental Materials. 1974 W.B. Saunders Co., Philadelphia, Seventh edition

11. Servais, G., Gartz, L. 1971, Structures of zinc phosphate dental cement. J. Dent. Res. 50: 613-620

12. Docking, A.R., Donnison, J.A., Newbury, C.R. and Storey, E. 1953, The effect of orthodontic cement on tooth enamel. Aust. J. Dent. 57: 139-142

13. Norman, R.D., Swartz, M.L., Phillips, R.W. and Raibley, J.W. 1966, Direct pH determination of setting cements. 1. A test method and the effects of storage time and media. J. Dent. Res. 45: 1214-1220

14. Norman, R.P., Swartz, M.L., Phillips, R.W. and Raiblez, J.W. 1966(b), Direct pH determination of setting cements 2. The effects of prolonged storage time, powder/liquid ratios, temperature and dentin. J. Dent. Res. 45: 1214-1216

15. Cook, F.W. and Horne, G.W. 1961, The acidity of zinc phosphate cement after setting. J. Dent. Res. 40: 624-630

16. Lefkowitz, W. 1940, Histological evidence of the harmful effect of cement under orthodontic bands. J. Dent. Res. 19: 47-52

17. Castello, I., Massler, M., Monteleone, U.L., and Suher, T. 1948, Effect of zinc oxyphosphate cement on enamel. Amer J. Ortho. 34: 271-275

18. Seniff, R.W. 1961, Change in enamel surface caused by oxyphosphate cement. J. Dent. Res. 40: 642-645

19. Muhler, J.C. 1958, The effect of a single topical application of stannous fluoride on incidence of dental carries in adults. J. Dent. Res. 37: 415-419

20. Gursin, A.V. 1965, Study of effect of stannous fluoride incorporated in dental cement. J. Oral Ther. 1: 630-636

21. Scull, C.E. 1970, A study of the incorporation of stannous fluoride into zinc oxyphosphate cement to determine its effect on decalcification under orthodontic bands. M.S. Thesis, University of Texas Dental Branch

22. Ram, Z., Gedalia, I., and Reisstein, I. 1973, Effects of stannous fluoride additions to zinc phosphate cement. J. Dent. Res. 52: 1344-1350

23. Wei, S.H., Sierk, D.L. 1971, Fluoride uptake by enamel from zinc phosphate cement containing stannous fluoride. JADA 83: 621-631

24. Myers, C.L., Long, R.P., Balser, J.D., Stookey, G.K. 1973, In vivo alterations in enamel from zinc phosphate cement containing stannous fluoride. JADA 87: 1216-1221

25. Sadowsky, P.L., Retief, D.H., and Bradley, E.L. 1981, Enamel fluoride uptake from orthodontic cements and its effect on demineralization. An. J. Orthod. 79(5): 523-533

26. Sadowsky, P.L., Retief, D.H., and Bradley, E.L. 1983, Acid resistance of enamel exposed to fluoride-containing orthodontic cements. Am J. Ortho 83(1): 33-37

27. McLean, J.W., and Wilson, A.D. 1977, The clinical development of the glass ionomer cements. formulation and preparation. Aust. Dent. J. 22(1): 31-36

28. Wilson, A.D. 1977, The development of glass ionomer cement. Dent. Update 4(7): 401 - 412

29. Crisp, S. and Wilson, A.D. 1974, Reactions in glass ionomer cements. Decomposition of the powder. J. Dent. Res. 53(6): 1408 - 1413

30. Crisp, S., et al 1974, Reactions in glass ionomer cements. An infared spectroscopic study. J. Dent. Res. 53(6): 1408 - 1413

31. Crisp, S. and Wilson, A.D. 1974, Reactions in glass ionomer cements. The precipation reaction. J. Dent. Res. 53(6): 1420-1424

32. Zumstein, Th. A. 1983, Adhesion of Cement. "Quintessence Inter. 4: 465 - 472

33. Wilson, A.D. 1975, Adhesion of glass ionomer cements in dentisitry. Aspects of adhesion. Transcipter Books,285-306

34. McLean, J.W. and Sced, I.R. 1977, A new method of bonding dental cements and porcelain to metal surfaces. Oper Dent. 2: 130 - 142

35. Powis, D.R., Folleras, T., Mierson, S.A. and Wilson, A.D. 1982, Improved adhesion of a glass ionomer cement to dentin and enamel. J. Dent. Res. 12: 1416 - 1422

36. McLean, J.W. and Wilson, A.D. 1977, The clinical development of the glass ionomer cement. Some clinical applications. Aust Dent. J. 22(2): 120-127

37. Kidd, E.A. 1978, Cavity sealing ability of composite and glass ionomer cement restorations. An assessment in vitro. Br. Dent. J. 144 (5): 139-142

38. Tveit, A.B. and Gjerdet, N.R. 1981, Fluoride release from a fluoride containing amalgam, a glass ionomer cement and a silicate cement. J. O. Rehab. 8(3): 237-241

39. Cranfield, M. 1982, Factors relating to the rate of fluoride-ion release from glass ionomer cement. J. of Dent. 10(4): 333-341

40. Sylvia, D., Mergon, S.D. and Smith, A.F. 1984, A comparison of fluoride release from three glass ionomer cements and a polycarboxylate cement. Int. End. Journ. 17(1): 124 - 127

41. Forstein, L. 1977, Fluoride release from a glass ionomer cement. Scand. J. Dent. Res. 85(6): 503-504

42. Retief, D.H., Bradley, E.L., Denton J.L. , and Switzer P. 1984, Enamel and cementum uptake fron a glass ionomer cement. Carries Res. 18: 250-257

43. De Fritas, J.F. 1968, The long term solubility of silicate cement. Aust. Dent. J. 13: 129 - 135

44. Michem, J.L., Gronu, D.G. 1981, Continued evaluation of the clinical solubility of luting cements. J. Pros Dent 45(3): 289 - 291

45. Causton, B.E. 1981, The Physio-mechanical consequences of exposing glass ionomer cements to water during setting. Biomaterials 2(2): 112 - 115

46. Oilo, G. 1981, Bond strength of new ionomer cements to dentin. Scad J. Dent. Res. 89(4): 344-347

47. Wilson, A.D., Crisp, S., Ferner, A.J. 1976, Reactions in glass-ionomer cements IV. Effect of chelating comonomers on setting behavior. J. Dent. Res. 55(3): 489-495

48. Pluim, L.J. et al. 1984, Qualitative cement solubility experiments in vivo. J. Oral Rehabil. 11(2): 171-179

49. Mount, G. 1982, The use of glass ionomer in clinical practice. Dental Outlook 8(3): 37-42

50. Phillips, R.W., Swartz, M.L. 1948, Effects of fluorides on hardness of tooth enamel. JADA 37: 1-25

51. Grieve, A.R. 1979, The production of carries-like leisions in vitro. Carries Res. 12: 35-49

52. American Dental Association, 1979. Status report on the glass ionomer cements. Prepared by J.W. McLean for the Council on Dental Materials and Devices. JADA 99: 221-226

53. Fehr, F.H. von der 1965, Maturation and remineralization of enamel (abstract only). Advances Fluorine Res. 3: 83-98

54. Adams, R. J. 1967, The effect of fixed orthodontic appliances on the cariogenicity, qualtity and microscopic morphology of oral lactobacilli. J. Oral Med. 22: 88-99

55. Bloom, R.H. and Brown, L.R. 1964, Study of the effect of orthodontic appliances on the oral microbial flora. Orqal Surg, Oral Med. and Oral Path. 17: 658-667

56. Coury, T.L., Wilker, R.D., Muconda, F.J., Probst, R.T. 1982, Adhesiveness of glass ionomer to enamel and dentin, a laboratory study. Oper Dent. 7: 2-6

57. Caryle, T.D., Chamma, A., Moir, R.W., Williams, P.T. 1978, An evaluation of the shear bond strength developed between a glass ionomer cement and enamel. J. Dent. Res. 57: 232

TABLE I.
TEETH CEMENTED WITH GLASS IONOMER, BEFORE VRS AFTER

TOOTH #	BEFORE LACTIC ACID CYCLING				AFTER CYCLING			DIFF SQR
	OBSER	SUM	AVER	DIFF	OBSER	SUM	AVER	
1B	4	4	1	0	4	4	1	0
2B	4	4	1	0	4	4	1	0
3A	4	4	1	0	4	4	1	0
4B	4	4	1	-0.25	4	5	1.25	0.0625
5B	4	4	1	0	4	4	1	0
6B	4	4	1	0	4	4	1	0
7B	4	4	1	0	4	4	1	0
8A	4	5	1.25	0.25	4	4	1	0.0625
9B	4	4	1	0	4	4	1	0
10A	4	4	1	0	4	4	1	0
11A	4	4	1	0	4	4	1	0
12A	4	4	1	-0.25	4	5	1.25	0.0625
13B	4	4	1	-0.25	4	5	1.25	0.0625
14B	4	4	1	0	4	4	1	0
15A	4	4	1	-0.25	4	5	1.25	0.0625
16B	4	4	1	-0.25	4	5	1.25	0.0625
17B	4	7	1.75	-0.25	4	8	2	0.0625
18B	4	4	1	0	4	4	1	0
19B	4	4	1	0	4	4	1	0
20A	4	5	1.25	0.25	4	4	1	0.0625
21B	4	4	1	-1	4	8	2	1
22A	4	4	1	0	4	4	1	0
23B	4	4	1	0	4	4	1	0
24A	4	4	1	-0.25	4	5	1.25	0.0625
25B	4	4	1	0	4	4	1	0
26B	4	4	1	0	4	4	1	0
27A	4	4	1	-0.25	4	5	1.25	0.0625
28B	4	4	1	0	4	4	1	0
29A	4	4	1	0	4	4	1	0
30B	4	4	1	0	4	4	1	0
31B	4	4	1	0	4	4	1	0
32B	4	4	1	0	4	4	1	0
33A	4	4	1	0	4	4	1	0
34A	4	4	1	0	4	4	1	0
35A	4	4	1	0	4	4	1	0
36B	4	4	1	-0.25	4	5	1.25	0.0625
37B	4	4	1	0	4	4	1	0
38B	4	5	1.25	0.25	4	4	1	0.0625
39A	4	4	1	0	4	4	1	0
40A	4	4	1	0	4	4	1	0
TOTALS	160	166	1.04	-2.50	160	176	1.10	1.75
SQUARE			1.08	6.25			1.21	
AVERAGE DIFF.				0.06				
STANDARD DEV.			0.13	0.20			0.23	

```
SUM OF DIFF.     -2.5
(DIFF) SQR       6.25
SUM (DIFF) SQR   1.75
M =              40      SQRT M = 6.324
STND DEV =       0.20
PAIRED T TEST    1.95 (not sig at the .01 level)
```

TABLE II.
TEETH CEMENTED WITH ZINC PHOSPHATE BEFORE VRS AFTER

| TOOTH # | BEFORE LACTIC ACID CYCLING | | | | AFTER CYCLING | | | |
	OBSER	SUM	AVER	DIFF	OBSER	SUM	AVER	DIFF SQR
1A	4	4	1	0	4	4	1	0.00
2A	4	4	1	-1	4	8	2	1.00
3B	4	4	1	-1	4	8	2	1.00
4A	4	4	1	-1.5	4	10	2.5	2.25
5A	4	4	1	-2	4	12	3	4.00
6A	4	4	1	-1	4	8	2	1.00
7A	4	4	1	-2.25	4	13	3.25	5.06
8B	4	4	1	-2.25	4	13	3.25	5.06
9A	4	4	1	-1	4	8	2	1.00
10B	4	4	1	-1	4	8	2	1.00
11B	4	4	1	-1	4	8	2	1.00
12B	4	4	1	-1	4	8	2	1.00
13A	4	4	1	-1.75	4	11	2.75	3.06
14A	4	4	1	-1.25	4	9	2.25	1.56
15B	4	4	1	-2.25	4	13	3.25	5.06
16A	4	4	1	-1.25	4	9	2.25	1.56
17A	4	4	1	-1	4	9	2	1.00
18A	4	4	1	-2.5	4	14	3.5	6.25
19A	4	4	1	-0.75	4	7	1.75	0.56
20A	4	4	1	-1	4	8	2	1.00
21B	4	4	1	-2	4	12	3	4.00
22B	4	8	2	-2	4	16	4	4.00
23A	4	4	1	-2	4	12	3	4.00
24B	4	4	1	-1	4	8	2	1.00
25A	4	8	2	0	4	8	2	0.00
26A	4	4	1	-2	4	12	3	4.00
27B	4	4	1	-2.5	4	14	3.5	6.25
28A	4	4	1	-1.75	4	11	2.75	3.06
29B	4	4	1	-1.25	4	9	2.25	1.56
30A	4	4	1	-2.25	4	13	3.25	5.06
31A	4	4	1	-1.5	4	10	2.5	2.25
32A	4	4	1	-0.75	4	7	1.75	0.56
33B	4	4	1	-2	4	12	3	4.00
34B	4	4	1	-0.75	4	7	1.75	0.56
35B	4	4	1	-1	4	8	2	1.00
36A	4	4	1	-0.25	4	5	1.25	0.06
37A	4	4	1	-1	4	8	2	1.00
38A	4	4	1	-2	4	12	3	4.00
39B	4	4	1	-2.25	4	13	3.25	5.06
40B	4	4	1	-0.75	4	7	1.75	0.56

TOTALS	160	168	1.05	55.75	160	392	2.44	95.44
SQUARE			1.10	3108			5.97	9108
AVERAGE DIFF.				1.394				
STANDARD DEV.			0.22	0.67			0.67	
SUM OF DIFF.		55.70		0.158				
(DIFF) SQR		3108.1						
SUM (DIFF) SQR		95.44						
M =		40						
STND DEV =		0.67						
PAIRED T TEST		13.07	(sig. at the .01 level)					

TABLE III.
GLASS IONOMER BEFORE VRS ZINC PHOSPHATE BEFORE

	GLASS IONOMER					ZINC PHOSPHATE			
TOOTH #	OBS.	SUM	AVER	DIFF.	TOOTH	OBS.	SUM	AVER.	DIFF SQR
1B	4	4	1	0	1A	4	4	1	0
2B	4	4	1	0	2A	4	4	1	0
3A	4	4	1	0	3B	4	4	1	0
4B	4	4	1	0	4A	4	4	1	0
5B	4	4	1	0	5A	4	4	1	0
6B	4	4	1	0	6A	4	4	1	0
7B	4	4	1	0	7A	4	4	1	0
8A	4	5	1.25	0.25	8B	4	4	1	0.0625
9B	4	4	1	0	9A	4	4	1	0
10A	4	4	1	0	10B	4	4	1	0
11A	4	4	1	0	11B	4	4	1	0
12A	4	4	1	0	12B	4	4	1	0
13B	4	4	1	0	13A	4	4	1	0
14B	4	4	1	0	14A	4	4	1	0
15A	4	4	1	0	15B	4	4	1	0
16B	4	4	1	0	16A	4	4	1	0
17B	4	7	1.75	0.75	17A	4	4	1	0.5625
18B	4	4	1	0	18A	4	4	1	0
19B	4	4	1	0	19A	4	4	1	0
20A	4	5	1.25	0.25	20A	4	4	1	0.0625
21B	4	4	1	0	21B	4	4	1	0
22A	4	4	1	-1	22B	4	8	2	1
23B	4	4	1	0	23A	4	4	1	0
24A	4	4	1	0	24B	4	4	1	0
25B	4	4	1	-1	25A	4	8	2	1
26B	4	4	1	0	26A	4	4	1	0
27A	4	4	1	0	27B	4	4	1	0
28B	4	4	1	0	28A	4	4	1	0
29A	4	4	1	0	29B	4	4	1	0
30B	4	4	1	0	30A	4	4	1	0
31B	4	4	1	0	31A	4	4	1	0
32B	4	4	1	0	32A	4	4	1	0
33A	4	4	1	0	33B	4	4	1	0
34A	4	4	1	0	34B	4	4	1	0
35A	4	4	1	0	35B	4	4	1	0
36B	4	4	1	0	36A	4	4	1	0
37B	4	4	1	0	37A	4	4	1	0
38B	4	5	1.25	0.25	38A	4	4	1	0.0625
39A	4	4	1	0	39B	4	4	1	0
40A	4	4	1	0	40B	4	4	1	0

```
TOTALS            160      166  1.03    -0.5         16  168    1.050       2.75
SQUARE                          1.07     0.250                  1.103
AVERAGE DIFFERENCE                       0.0125
STANDARD DEVIATION              0.13     0.262                  0.218
SUM OF DIFFERENCE    -0.5
SUM OF DIFF SQR       0.25
SUM OF SQR OF DIF     2.75
M =                   40  SQRT 6.32
STND DEV =            0.265
PAIRED T TEST      0.29    (not sig. at .01 level)
```

TABLE IV. GLASS IONOMER AFTER VRS ZINC PHOSPHATE AFTER

| | GLASS IONOMER | | | | | ZINC PHOSPHATE | | | DIFF |
TOOTH #	OBSER	SUM	AVER	DIFF	TOOTH	OBS	SUM	AVER.	SQR
1B	4	4	1	0	1A	4	4	1	0
2B	4	4	1	-1	2A	4	8	2	1
3A	4	4	1	-1	3B	4	8	2	1
4B	4	5	1.25	-1.25	4A	4	10	2.5	1.5625
5B	4	4	1	-2	5A	4	12	3	4
6B	4	4	1	-1	6A	4	8	2	1
7B	4	4	1	-2.25	7A	4	13	3.25	5.0625
8A	4	4	1	-2.25	8B	4	13	3.25	5.0625
9B	4	4	1	-1	9A	4	8	2	1
10A	4	4	1	-1	10B	4	8	2	1
11A	4	4	1	-1	11B	4	8	2	1
12A	4	5	1.25	-0.75	12B	4	8	2	0.5625
13B	4	5	1.25	-1.5	13A	4	11	2.75	2.25
14B	4	4	1	-1.25	14A	4	9	2.25	1.5625
15A	4	5	1.25	-2	15B	4	13	3.25	4
16B	4	5	1.25	-1	16A	4	9	2.25	1
17B	4	8	2	0	17A	4	9	2	0
18B	4	4	1	-2.5	18A	4	14	3.5	6.25
19B	4	4	1	-0.75	19A	4	7	1.75	0.5625
20A	4	4	1	-1	20A	4	8	2	1
21B	4	8	2	-1	21B	4	12	3	1
22A	4	4	1	-3	22B	4	16	4	9
23B	4	4	1	-2	23A	4	12	3	4
24A	4	5	1.25	-0.75	24B	4	8	2	0.5625
25B	4	4	1	-1	25A	4	8	2	1
26B	4	4	1	-2	26A	4	12	3	4
27A	4	5	1.25	-2.25	27B	4	14	3.5	5.0625
28B	4	4	1	-1.75	28A	4	11	2.75	3.0625
29A	4	4	1	-1.25	29B	4	9	2.25	1.5625
30B	4	4	1	-2.25	30A	4	13	3.25	5.0625
31B	4	4	1	-1.5	31A	4	10	2.5	2.25
32B	4	4	1	-0.75	32A	4	7	1.75	0.5625
33A	4	4	1	-2	33B	4	12	3	4
34A	4	4	1	-0.75	34B	4	7	1.75	0.5625
35A	4	4	1	-1	35B	4	8	2	1
36B	4	5	1.25	0	36A	4	5	1.25	0
37B	4	4	1	-1	37A	4	8	2	1
38B	4	4	1	-2	38A	4	12	3	4
39A	4	4	1	-2.25	39B	4	13	3.25	5.0625
40A	4	4	1	-0.75	40B	4	7	1.75	0.5625
TOTALS	160	176	1.1	53.75		16	392	2.444	92.1875
SQUARE			1.21	2889.0				5.972	
AVERAGE DIFF.				1.34					
STANDARD DEV.			0.22	0.71				0.672	

SUM OF DIFF.   53.75
(DIFF) SQR   2889.0
SUM (DIFF) SQR   92.18
M =   40   SQRT 6.324
STND DEV =   0.71
PAIRED T TEST   11.87 (sig at .01 level)

TABLE V. COMPARISON OF SUBSET TO TOTAL SETS

SET OF 30 VRS SET OF 40 GLASS IONOMER BEFORE

	30			40		
SUM	AVER.	S.D.	SUM	AVER.	S.D.	DIFFERENCE
126	1.05	0.20	166	1.04	0.20	0.01

SET OF 30 VRS SET OF 40 GLASS IONOMER AFTER

	30			40		
SUM	AVER.	S.D.	SUM	AVER.	S.D.	DIFFERENCE
135	1.12	0.20	176	1.10	0.20	0.03

SET OF 30 VRS SET OF 40 ZINC PHOSPHATE BEFORE

	30			40		
SUM	AVER.	S.D.	SUM	AVER.	S.D.	DIFFERENCE
128	1.07	0.66	168	1.05	0.66	0.02

SET OF 30 VRS SET OF 40 ZINC PHOSPHATE AFTER

	30			40		
SUM	AVER.	S.D.	SUM	AVER.	S.D.	DIFFERENCE
295	2.45	0.66	392	2.44	0.66	0.01

TABLE VII. CA++ ION AND pH DETERMINATION OF TEN MATCHED SETS

TEETH #	Ca++	pH 1	pH 2	Av. pH	Ca++ DIFF	pH 1 DIFF	pH 2 DIFF
3A G	0.14	5.30	5.22	5.26	0.18	0.74	0.26
3B Z	0.32	6.04	5.50	5.77			
4A Z	0.36	5.50	5.80	5.65	0.35	0.40	0.17
4B G	0.01	5.10	5.53	5.32			
5A Z	0.34	5.68	5.50	5.59	0.32	0.78	0.39
5B G	0.02	4.90	5.11	5.01			
6A Z	0.35	5.61	5.75	5.68	0.16	0.38	0.45
6B G	0.19	5.23	5.30	5.27			
10A G	0.18	5.20	4.54	4.87	0.13	0.12	0.53
10B Z	0.31	5.08	5.07	5.08			
28A Z	0.35	5.91	5.45	5.68	0.24	0.86	0.46
28B G	0.11	5.05	4.99	5.02			
31A Z	0.35	6.30	5.92	6.11	0.28	1.06	0.86
31B G	0.07	5.24	5.06	5.15			
36A Z	0.34	5.82	5.75	5.79	0.20	0.60	0.57
36B G	0.14	5.22	5.18	5.20			
39A G	0.17	4.76	4.78	4.77	0.18	1.09	0.74
39B Z	0.35	5.85	5.52	5.69			
40A G	0.05	4.98	4.78	4.88	0.06	0.78	0.99
40B Z	0.11	5.76	5.77	5.77			
SUMS	4.26	108.53	106.	107.53	2.10	6.81	5.42
AVERAGE	0.21	5.43	5.33	5.38	0.21	0.68	0.54
STND DEV.	0.12	0.41	0.38	0.37	0.08	0.29	0.24
PAIRED T					7.88	7.36	6.99

(all sig. at .01 level)

TABLE VIII.
OBSERVER ERRORS BETWEEN 1ST AND 2ND DECALCIFICATION READINGS

GLASS IONOMER			ZINC PHOSPHATE			
BEFORE	AFTER	TOTAL	BEFORE	AFTER	TOTAL	GRAND TOTAL
2	7	9	3	22	25	34

AV. ERROR (%)

1.25%	4.38%	3.44%	1.88%	14.0%	15.63%	7.8%

TOTAL OBSERVATION DIFFERENCES   5.32%
   (ALL DIFFERENCES WERE ONE SCALE INCREMENTS)

SCALE:

   1 = NO COLOR DIFFERENCES

   2 = SLIGHT COLOR DIFFERENCES

   3 = WHITENING

   4 = DISCOLORATION WITH ROUGHNESS

   5 = CARRIES

TABLE IX. CALCIUM ION CONCENTRATION AND AVERAGE PH

|	GLASS IONOMER		ZINC PHOSPHATE				
TEETH #	CA++	Av. pH	#	[Ca++]	Av.pH	Ca++ DIF	PH DIFF
3A	0.14	5.26	3B	0.32	5.77	-0.1	-0.5
4B	0.01	5.32	4A	0.36	5.65	-0.3	-0.3
5B	0.02	5.01	5A	0.34	5.59	-0.3	-0.5
6B	0.19	5.27	6A	0.35	5.68	-0.1	-0.4
10A	0.18	4.87	10B	0.31	5.08	-0.1	-0.2
28B	0.11	5.02	28A	0.35	5.68	-0.2	-0.6
31B	0.07	5.15	31A	0.35	6.11	-0.2	-0.9
36B	0.14	5.20	36A	0.34	5.79	-0.2	-0.5
39A	0.17	4.77	39B	0.35	5.69	-0.1	-0.9
40A	0.05	4.88	40B	0.11	5.77	-0.0	-0.8
SUMS	1.08	50.75		3.18	56.8	-2.1	-6.0
AVERAGES	0.11	5.08		0.32	5.68	0.21	0.61
VARIANCE	.00	0.03		0.01	0.06	0.01	0.06
STND DEV.	0.06	0.18		0.07	0.24	0.08	0.24
PAIRED T					7.88	7.89	

(all sig. at .01 level)
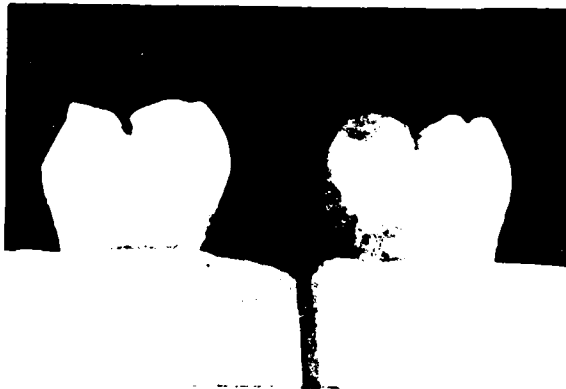
FIG. 1. Matched teeth in acrylic blocks ready for mounting.

FIG. 2. Examples of enamel surface changes.

A. NONE: No change, normal surface
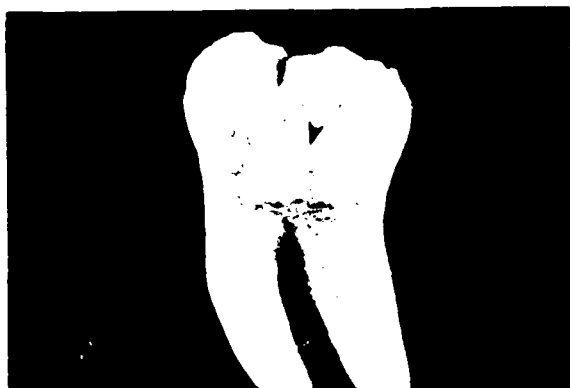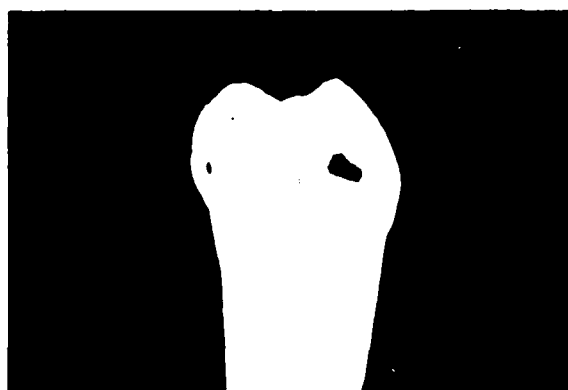
B. MILD: Slight change in enamel color (at arrow).

FIG. 2
C. MODERATE: Definate whitening of enamel without break in continuity of enamel color (at arrow).

D. SEVERE: Obvious area of discoloration with roughness and break in continuity of surface (at arrow).
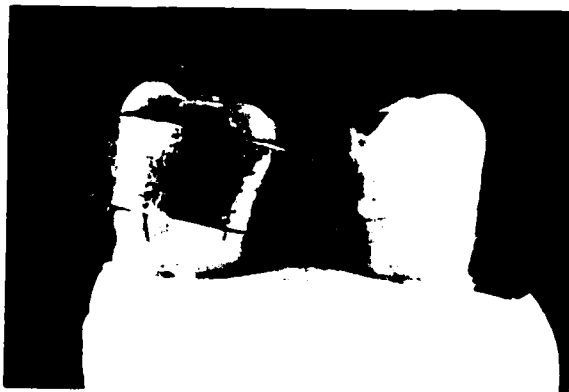

E. CARIES: Obvious break in continuity of enamel surface.

FIG. 3. Banded tooth with
control after acid cycling;
note, complete decalcifi-
cation of non-banded control
tooth.

FIG. 4. Glass ionomer (G)
tooth sample with control (C)
after decalcification; note,
adherence of the glass ionomer
cement to the surface of the
debanded sample (at arrow).

FIG. 7. Tooth sample cemented
with zinc phosphate cement; not
indistinct border between bande
area and non-banded area (arrow
Compare these surfaces with the
glass ionomer cemented samples.
(fig. 8.)

A. buccal

FIG 7. B. Lingual

FIG. 7. C. Mesial

FIG. 7. D. Distal

FIG. 8. Matching tooth from
fig. 7. cemented with glass
ionomer cement; note, in banded
areas, the glassy surfaces and
the distinct borders between
banded and non-banded areas
(arrows).

A. Buccal
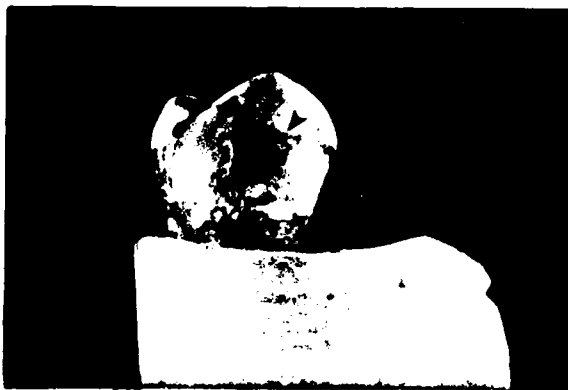
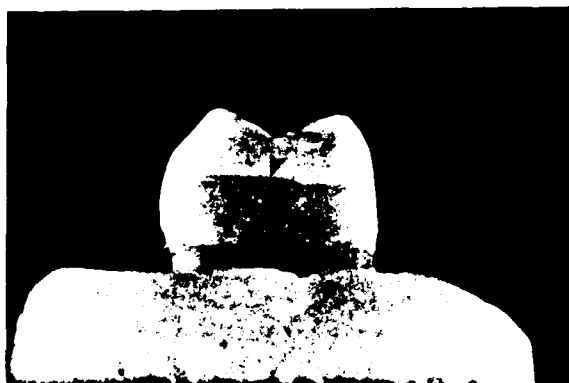B. Lingual

FIG. 8. C. Mesial
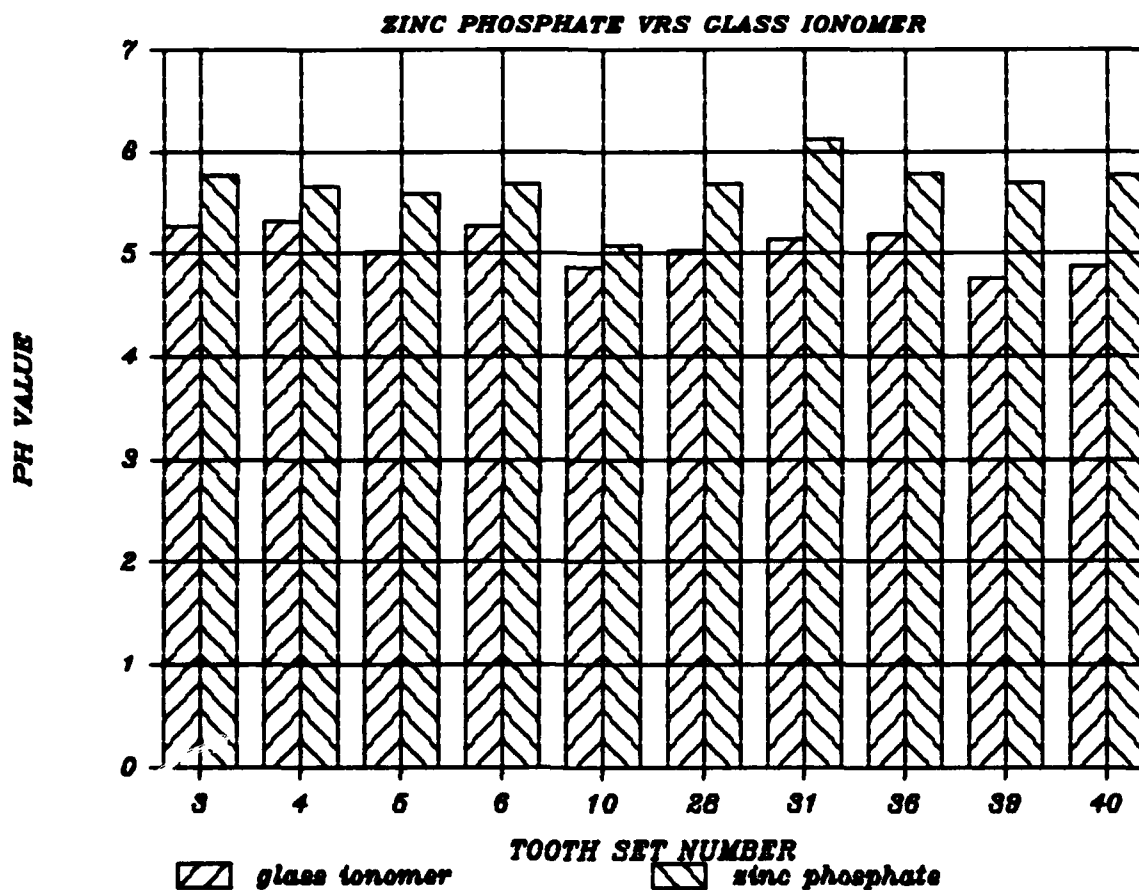
FIG. 8. D. Distal
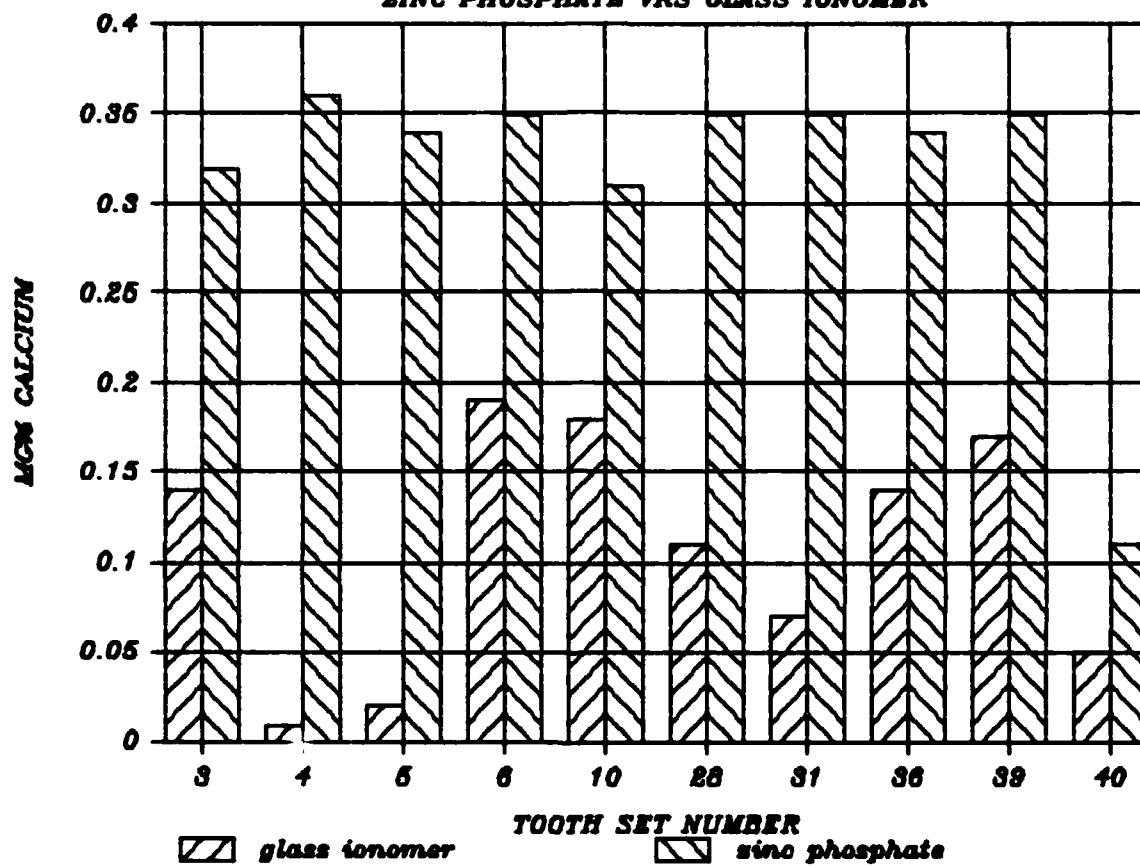
FIG 5. AVERAGE PH OF TEST SOLUTIONS

ZINC PHOSPHATE VRS GLASS IONOMER

PH VALUE

TOOTH SET NUMBER

glass ionomer   zinc phosphate

FIG 6. CALCIUM ION CONCENTRATION

ZINC PHOSPHATE VRS GLASS IONOMER

FISHER SPECTROPHOTOMETRIC CALCIUM DETERMINATION METHOD

1. Prepare working standards as follows from Fisher's 15mg% stock.

Standard	ml distilled water	mg% final conc.
0 ml	0.3 ml	blank
0.1 ml	0.2 ml	5 mg%
0.2 ml	0.1 ml	10mg%
0.3 ml	0 ml	15mg%

2. Mark test tubes as follows: B (blank): 5S, 10S, and 15S (standards). C (controls), and numbers for unknowns.

3. Pipette 0.1 ml of each sample directly into the bottom of the correspondingly marked test tube

4. Add 3.0 ml of cresolphtlaein complexone (CS-952A) to each tube, and mix well.

5. Add 3.0 ml of diethylamine (CS-952B) to each tube, and mix well.

6. Incubate all tubes at room temperature (20-30°C) for 5 minutes.

7. Read the absorbance of each sample against the reagent blank set a zero absorbance at 540nm.

8. Plot the absorbance of each standard (5, 10, and 15 mg%) against its concentration. Read the values for all unknowns directly from the graph.

9. Results are expressed as mg% (mg/dl).

END

FILMED

12-85

DTIC